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Ethyl (*E*)-4-(2-acetylphenoxy)but-2-enoate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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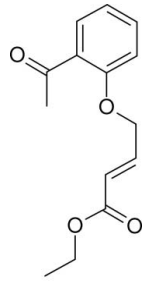
The crystal packing in the title compound, $C_{14}H_{16}O_4$, is controlled by van der Waals interactions.

Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies of cyclization reactions (Williamson *et al.*, 2007). The dihedral angles between the mean plane of the C3–C8 benzene ring and the planes of the C1/C2/O1 and the C12/O3/O4 groups are 4.26 (6) and 9.63 (12)°, respectively. The C11—C12 bond length of 1.4787 (16) Å implies that there is little, if any, delocalization of electrons between the C12/O3/O4 and C10/C11 groups in the side chain. A similar result was found for the equivalent bond in ethyl (*E*)-4-(2-formylphenoxy)but-2-enoate (Williamson *et al.*, 2005). Otherwise, the geometrical parameters for (I) may be regarded as normal (Allen *et al.*, 1987).



(I)

There are no clear-cut directional intermolecular bonding interactions in the crystal structure of (I). The minimum separation of the centroids of the benzene rings of nearby molecules is greater than 5.4 Å.

Experimental

A dry two-necked flask was charged with NaH (0.360 g, 15 mmol) and washed with dry petrol (3 × 1 ml). Dry DMF (40 ml) was added, and the suspension cooled to 273 K. 2-Hydroxyacetophenone (1.361 g, 1.20 ml, 10 mmol) was added and the solution stirred for 20 min. Ethyl 4-bromocrotonate (2.82 g, 2.01 ml, 11 mmol) was added in one portion. The solution was allowed to warm to room temperature, and stirred for 18 h. H_2O (60 ml) was added, followed by extraction with Et_2O (3 × 50 ml). The combined organics were washed with saturated brine (75 ml), dried over $MgSO_4$, and the solvent removed *in vacuo*. Chromatographic elution with 20% EtOAc in hexane, and collecting the fraction with R_F = 0.22 yielded the desired product as colourless needles (1.263 g, 51%), which were recrystallized from EtOH; analysis calculated for $C_{14}H_{16}O_4$: C 67.73, H 6.50%; found C 67.61, H 6.59%;

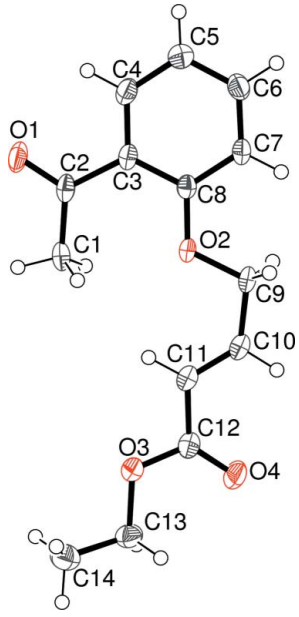


Figure 1
View of the molecular structure of (I), showing 50% displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radius.

Crystal data

$C_{14}H_{16}O_4$
M_r = 248.27
Monoclinic, $C2/c$
a = 23.7480 (8) Å
b = 7.2686 (3) Å
c = 15.8710 (4) Å
β = 112.2400 (13)°
V = 2535.76 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 (2) K
0.48 × 0.42 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.956, T_{max} = 0.978
25327 measured reflections
2909 independent reflections
2396 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.06
2909 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip, to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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supporting information

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Crystal data

$C_{14}H_{16}O_4$
$M_r = 248.27$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 23.7480\ (8)$ Å
$b = 7.2686\ (3)$ Å
$c = 15.8710\ (4)$ Å
$\beta = 112.2400\ (13)°$
$V = 2535.76\ (15)$ Å^3
$Z = 8$
$F(000) = 1056$
$D_x = 1.301$ Mg m^{-3}
Melting point = 325–327 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5501 reflections
$\theta = 2.9–27.5°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Block, colourless
0.48 × 0.42 × 0.24 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 1999)
$T_{min} = 0.956$, $T_{max} = 0.978$
25327 measured reflections
2909 independent reflections
2396 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -30\rightarrow30$
$k = -9\rightarrow8$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.105$
$S = 1.06$
2909 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.9651P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0042 (10)

Special details

Experimental. ν_{max}(KBr)/cm^{-1} 2971 (Ar), 2893 [C=O (aldehyde)], 1704 [C=O (ester)], 1646 [C=O (aldehyde)].

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.22000 (5)	0.97136 (16)	−0.10660 (7)	0.0291 (3)
H1A	0.2045	0.9573	−0.1729	0.044*
H1B	0.1934	1.0545	−0.0900	0.044*
H1C	0.2210	0.8510	−0.0783	0.044*
C2	0.28295 (5)	1.04987 (14)	−0.07388 (7)	0.0256 (3)
C3	0.31980 (5)	1.07664 (14)	0.02575 (7)	0.0237 (2)
C4	0.37937 (5)	1.13971 (16)	0.04821 (8)	0.0301 (3)
H4	0.3939	1.1595	0.0008	0.036*
C5	0.41776 (6)	1.17410 (18)	0.13700 (8)	0.0343 (3)
H5	0.4578	1.2186	0.1503	0.041*
C6	0.39703 (5)	1.14285 (16)	0.20640 (8)	0.0305 (3)
H6	0.4232	1.1656	0.2677	0.037*
C7	0.33858 (5)	1.07876 (15)	0.18729 (7)	0.0255 (2)
H7	0.3250	1.0569	0.2355	0.031*
C8	0.29952 (5)	1.04603 (14)	0.09738 (7)	0.0219 (2)
C9	0.22103 (5)	0.95766 (16)	0.14867 (7)	0.0261 (3)
H9A	0.2231	1.0753	0.1811	0.031*
H9B	0.2481	0.8682	0.1924	0.031*
C10	0.15761 (5)	0.88752 (15)	0.11317 (7)	0.0271 (3)
H10	0.1412	0.8564	0.1574	0.033*
C11	0.12123 (5)	0.86334 (15)	0.02712 (7)	0.0275 (3)
H11	0.1356	0.8877	−0.0201	0.033*
C12	0.05817 (5)	0.79840 (15)	0.00437 (8)	0.0285 (3)
C13	−0.03758 (5)	0.75614 (18)	−0.11551 (9)	0.0345 (3)
H13A	−0.0562	0.8164	−0.0765	0.041*
H13B	−0.0420	0.6213	−0.1117	0.041*
C14	−0.06821 (6)	0.8193 (2)	−0.21187 (9)	0.0422 (3)
H14A	−0.1118	0.7926	−0.2332	0.063*
H14B	−0.0507	0.7546	−0.2504	0.063*
H14C	−0.0622	0.9521	−0.2152	0.063*
O1	0.30498 (4)	1.09337 (13)	−0.12924 (5)	0.0368 (2)
O2	0.24104 (3)	0.98547 (10)	0.07529 (5)	0.0247 (2)
O3	0.02650 (4)	0.80537 (11)	−0.08531 (5)	0.0317 (2)
O4	0.03676 (4)	0.74967 (14)	0.05879 (6)	0.0431 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0401 (7)	0.0312 (6)	0.0162 (5)	0.0016 (5)	0.0107 (5)	0.0001 (4)
C2	0.0370 (6)	0.0242 (5)	0.0189 (5)	0.0078 (4)	0.0143 (5)	0.0023 (4)
C3	0.0312 (6)	0.0232 (5)	0.0195 (5)	0.0051 (4)	0.0127 (4)	0.0025 (4)
C4	0.0342 (6)	0.0352 (6)	0.0262 (6)	0.0024 (5)	0.0173 (5)	0.0030 (5)
C5	0.0281 (6)	0.0435 (7)	0.0318 (6)	−0.0007 (5)	0.0119 (5)	0.0015 (5)
C6	0.0306 (6)	0.0360 (6)	0.0217 (5)	0.0014 (5)	0.0062 (5)	0.0006 (5)
C7	0.0319 (6)	0.0282 (6)	0.0175 (5)	0.0035 (4)	0.0107 (4)	0.0024 (4)
C8	0.0266 (5)	0.0207 (5)	0.0193 (5)	0.0038 (4)	0.0097 (4)	0.0017 (4)
C9	0.0342 (6)	0.0303 (6)	0.0175 (5)	0.0006 (4)	0.0138 (4)	0.0016 (4)
C10	0.0343 (6)	0.0279 (5)	0.0247 (5)	0.0000 (5)	0.0174 (5)	0.0019 (4)
C11	0.0324 (6)	0.0292 (6)	0.0253 (5)	0.0000 (5)	0.0158 (5)	0.0012 (4)
C12	0.0353 (6)	0.0274 (6)	0.0256 (6)	−0.0019 (5)	0.0147 (5)	−0.0006 (4)
C13	0.0293 (6)	0.0383 (6)	0.0376 (7)	−0.0067 (5)	0.0146 (5)	−0.0022 (5)
C14	0.0332 (7)	0.0477 (8)	0.0414 (7)	−0.0044 (6)	0.0093 (6)	0.0013 (6)
O1	0.0454 (5)	0.0494 (5)	0.0221 (4)	0.0018 (4)	0.0201 (4)	0.0029 (4)
O2	0.0285 (4)	0.0325 (4)	0.0153 (4)	−0.0011 (3)	0.0108 (3)	0.0006 (3)
O3	0.0297 (4)	0.0412 (5)	0.0260 (4)	−0.0049 (3)	0.0126 (3)	0.0004 (3)
O4	0.0452 (5)	0.0591 (6)	0.0309 (5)	−0.0168 (5)	0.0212 (4)	0.0005 (4)

Geometric parameters (Å, º)

C1—C2	1.4974 (16)	C9—O2	1.4289 (12)
C1—H1A	0.9800	C9—C10	1.4844 (16)
C1—H1B	0.9800	C9—H9A	0.9900
C1—H1C	0.9800	C9—H9B	0.9900
C2—O1	1.2222 (13)	C10—C11	1.3225 (16)
C2—C3	1.5021 (14)	C10—H10	0.9500
C3—C4	1.3990 (16)	C11—C12	1.4787 (16)
C3—C8	1.4099 (14)	C11—H11	0.9500
C4—C5	1.3810 (17)	C12—O4	1.2093 (14)
C4—H4	0.9500	C12—O3	1.3355 (14)
C5—C6	1.3848 (16)	C13—O3	1.4564 (14)
C5—H5	0.9500	C13—C14	1.4956 (18)
C6—C7	1.3845 (16)	C13—H13A	0.9900
C6—H6	0.9500	C13—H13B	0.9900
C7—C8	1.3969 (14)	C14—H14A	0.9800
C7—H7	0.9500	C14—H14B	0.9800
C8—O2	1.3701 (13)	C14—H14C	0.9800
C2—C1—H1A	109.5	O2—C9—H9A	109.7
C2—C1—H1B	109.5	C10—C9—H9A	109.7
H1A—C1—H1B	109.5	O2—C9—H9B	109.7
C2—C1—H1C	109.5	C10—C9—H9B	109.7
H1A—C1—H1C	109.5	H9A—C9—H9B	108.2
H1B—C1—H1C	109.5	C11—C10—C9	127.59 (10)

O1—C2—C1	119.47 (10)	C11—C10—H10	116.2
O1—C2—C3	119.08 (10)	C9—C10—H10	116.2
C1—C2—C3	121.45 (9)	C10—C11—C12	120.00 (10)
C4—C3—C8	117.86 (9)	C10—C11—H11	120.0
C4—C3—C2	116.14 (9)	C12—C11—H11	120.0
C8—C3—C2	125.99 (10)	O4—C12—O3	123.56 (11)
C5—C4—C3	122.18 (10)	O4—C12—C11	125.43 (10)
C5—C4—H4	118.9	O3—C12—C11	110.98 (9)
C3—C4—H4	118.9	O3—C13—C14	107.72 (10)
C4—C5—C6	119.07 (11)	O3—C13—H13A	110.2
C4—C5—H5	120.5	C14—C13—H13A	110.2
C6—C5—H5	120.5	O3—C13—H13B	110.2
C7—C6—C5	120.64 (10)	C14—C13—H13B	110.2
C7—C6—H6	119.7	H13A—C13—H13B	108.5
C5—C6—H6	119.7	C13—C14—H14A	109.5
C6—C7—C8	120.28 (10)	C13—C14—H14B	109.5
C6—C7—H7	119.9	H14A—C14—H14B	109.5
C8—C7—H7	119.9	C13—C14—H14C	109.5
O2—C8—C7	122.21 (9)	H14A—C14—H14C	109.5
O2—C8—C3	117.82 (9)	H14B—C14—H14C	109.5
C7—C8—C3	119.96 (10)	C8—O2—C9	117.03 (8)
O2—C9—C10	109.98 (8)	C12—O3—C13	116.00 (9)

O1—C2—C3—C4	−4.09 (15)	C4—C3—C8—C7	0.02 (15)
C1—C2—C3—C4	175.94 (10)	C2—C3—C8—C7	−179.23 (10)
O1—C2—C3—C8	175.16 (10)	O2—C9—C10—C11	−4.35 (16)
C1—C2—C3—C8	−4.80 (16)	C9—C10—C11—C12	−177.61 (10)
C8—C3—C4—C5	−0.79 (17)	C10—C11—C12—O4	−6.17 (18)
C2—C3—C4—C5	178.53 (10)	C10—C11—C12—O3	171.96 (10)
C3—C4—C5—C6	0.92 (18)	C7—C8—O2—C9	0.38 (14)
C4—C5—C6—C7	−0.27 (18)	C3—C8—O2—C9	−179.16 (9)
C5—C6—C7—C8	−0.48 (17)	C10—C9—O2—C8	−178.45 (8)
C6—C7—C8—O2	−178.93 (10)	O4—C12—O3—C13	2.08 (17)
C6—C7—C8—C3	0.60 (16)	C11—C12—O3—C13	−176.09 (9)
C4—C3—C8—O2	179.57 (9)	C14—C13—O3—C12	165.19 (10)
C2—C3—C8—O2	0.32 (15)		